

FINAL REPORT

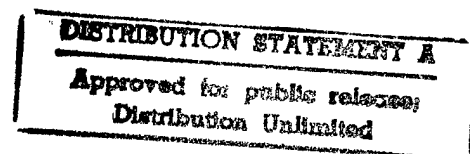
ARMY RESEARCH OFFICE GRANT 31117-MS

**TRANSMISSION ELECTRON HOLOGRAPHY
OF POLYMER MICROSTRUCTURE**

submitted by:

Professor Matthew R. Libera

Dept. of Materials Science and Engineering
Stevens Institute of Technology
Hoboken, NJ 07030



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13. ABSTRACT (Maximum 200 words)

This is the final report describing a four-year effort to develop and apply phase-contrast imaging based on transmission electron holography as a means to measure unstained multiphase polymer microstructure. The experiments involve a 200keV transmission electron microscope with a high-coherence field-emission electron source. A Mollenstadt biprism was constructed and installed to perform holographic interference experiments, and a slow-scan ccd camera was installed to provide for linear and fully digital hologram recording. A technique was developed to measure the average electron-optical refractive index characteristic of amorphous polymers. This method exploited the model spherical geometry of ~50nm diameter polymer latexes. This geometry enabled specimen thickness to be decoupled from the refractive effects of the material as measured by characteristic phase shifts imparted on an incident electron wave. The key quantity involved in the average refraction is known as the mean inner potential (MIP). The MIP for polystyrene was determined experimentally and agrees with predictions of simple models based on electrostatic properties of the polymer. Similar measurements were made on amorphous and crystalline silicon nanospheres yielding a MIP in excellent agreement with LDA calculations of Spence et al. at ASU. Simulations of holographic phase imaging of two-phase polymer microstructure were performed which indicate that the technique is sufficiently sensitive to distinguish between subtly different polymeric materials where no amplitude contrast can be observed.

14. SUBJECT TERMS
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I. OVERVIEW AND GOAL

This research concentrated on the development and application of phase-contrast imaging methods by transmission electron holography to study unstained multiphase polymer microstructure. Successful phase-contrast electron-optical imaging requires that different polymer phases be distinguished by different values of electron-optical refractive index. Little is known about the electron-refractive properties of polymers. Hence, the principal basic focus of this project has been to establish and use a novel method for quantifying electron refractive properties characteristic of polymers as well as select inorganic materials.

II. BACKGROUND

The positive Coulombic potential of a material accelerates an incident high-energy electron. The electron wavelength is shorter in the specimen, so the electron phase advances relative to an unmodulated vacuum reference wave. An average electron-optical refractive index, n_{eo} , can be described as:

$$n_{eo} = 1 + \frac{\Phi_0}{E} \frac{E_0 + E}{2E_0 + E} \quad [1]$$

where E is the accelerating potential and E_0 is the electron rest energy. Ignoring dynamical effects, a path difference $\Delta\phi$ relative to a vacuum reference wave can be defined for a wave passing through a specimen of thickness t :

$$\Delta\phi = 2\pi(n_{eo} - 1) t / \lambda \quad [2]$$

where λ is the relativistic electron wavelength. These equations can be combined to describe the phase shift in terms of Φ_0 and t :

$$\Delta\phi = \Delta\phi(t) = C_E \Phi_0 t \quad [3]$$

where $C_E = 2\pi(E_0 + E) / [\lambda E(2E_0 + E)]$ is a constant. At 200keV, $C_E = 7.312 \times 10^{-3}$ in units of (rad)/(V)(nm).

The phase shift $\Delta\phi(x,y)$ induced by refraction in a specimen relative to an unmodulated reference wave can be measured using

transmission electron holography. Holography was introduced by Gabor in the late 1940's to recover the entire exit-face wave function $\psi_e(x,y) = A_e(x,y)\exp[-\phi_e(x,y)]$ with both amplitude, $A_e(x,y)$, and phase, $\phi_e(x,y)$. Traditional imaging techniques measure only intensity $I(x,y) = |A(x,y)|^2$ and recover only amplitude information.

Holography is based on the coherent interference between an electron wave modulated by a specimen $\psi_e(x,y)$ and an unmodulated vacuum reference wave $\psi_r(x,y)$. An electron biprism can be used to combine the two waves. Because they are coherent, the waves interfere to give a cosine-type fringe pattern. Deviations in fringe position from a strictly cosine pattern carry image-wave phase information, and the fringe intensity carries image-wave amplitude information. Digital reconstruction of an electron hologram involves a series of steps: (i) Fourier transformation of the recorded hologram to generate a reciprocal-space pattern with an autocorrelation function plus two side bands; (ii) selection of one side band using a digital aperture; and (iii) recovery of real-space wave information by inverse Fourier transform of selected sideband data. The result is complex data describing the exit-face wavefunction, with both amplitude and phase, modulated by the transfer properties of the microscope.

Holography can be used directly to determine the mean inner potential provided that the thickness of the specimen is known. Φ_0 can be calculated using equation [3] given values for $\Delta\phi$ and t . This method has been exploited by Gajdardziska et al. using specimens with a known dependence of thickness on lateral position. Cleaved wedges were used to determine Φ_0 characteristic of Si, GaAs, MgO, and PbS.

The present research concentrated on the study of spherically shaped specimens. Like wedges, spheres provide a model shape with which to determine specimen thickness from lateral position in a two-dimensional image. Specimen thickness t is simply related to the lateral coordinates x and y by:

$$t = 2[R_0^2 - (x-x_0)^2 - (y-y_0)^2]^{1/2} \quad [4]$$

where (x_0, y_0) defines the sphere center in the 2-D image and R_0 is the sphere radius. Equation [4] is valid for the subset of x and y satisfying $(x-x_0)^2 + (y-y_0)^2 \leq R_0^2$. Equations [3] and [4] can

be combined to remove the t dependence of Φ_0 and describe the phase shift of an electron wave due to a spherical specimen as:

$$\Delta\phi = 2C_E \Phi_0 \{ [R_0^2 - (x-x_0)^2 - (y-y_0)^2]^{1/2} \} \quad [5]$$

The mean inner potential can be retrieved by measuring phase shift as a function of lateral position $\Delta\phi(x, y)$ if the parameters x_0 , y_0 , and R_0 are known. These can be established from the experimental data.

III. EXPERIMENTAL PROCEDURE **AND PROCEDURAL ACCOMPLISHMENTS**

This research was fundamentally enabled by the use of a transmission electron microscope at Stevens with a field-emission (FEG) electron source. The high brightness of this type of emitter provides sufficient coherence to do holographic interference experiments.

A significant experimental achievement early in the project was the design and construction of an electron biprism. This biprism was a necessary modification to the existing microscope. Its purpose is to controllably interfere an exit-face electron wave carrying information characteristic of a specimen with an unmodulated reference wave. The Stevens biprism was constructed in 1994 and used throughout subsequent and ongoing experiments. It is described by figure 1.

A second modification to the microscope, made in the fall of 1995, involved the acquisition of a slow-scan ccd camera system. Funds provided by ARO were leveraged to raise additional support through the New Jersey Commission on Science and Technology. This slow-scan camera enables acquisition of fully digital holograms with complete linearity of recorded intensity and accurate spatial alignment of images collected in series. All of these attributes are essential to the execution of fully quantitative holographic phase-imaging measurements.

In terms of methodology, the Stevens group innovated the use of spherical specimens which enable the intrinsic refractive effects of the specimen to be separated from the thickness effects on phase shift of an incident electron wave. Evaluation of experimental data collected from spherically-shaped specimens requires the simultaneous determination of the sphere's center

(x_0, y_0) , radius (R_0), and mean inner potential (Φ_0). The phase-image data sets evaluated often contained as many as 20,000 data points, and a numerical data-processing algorithm was developed based on a 4-parameter χ -squared method to establish the values of x_0 , y_0 , R_0 , and Φ_0 characteristic of each specimen. Simulations of the holographic phase-imaging experiment were performed which successfully validated the χ -squared software developed.

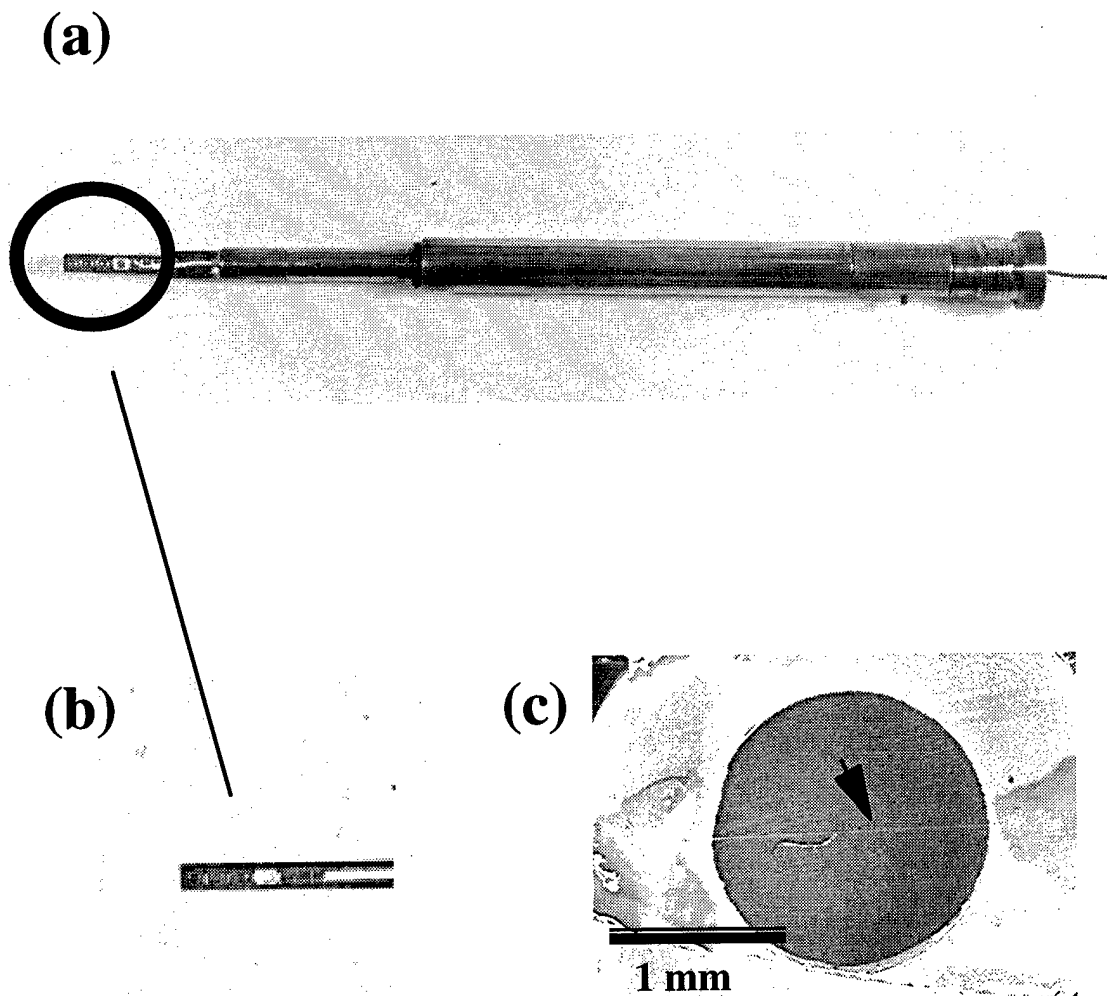


Fig. 1 - (a) modified SAD aperture rod with electrical feedthrough; (b) detail of aperture holder with three conventional SAD apertures; and (c) SEM image of the biprism filament (arrow) mounted on its insulating ceramic washer.

IV. RESULTS AND SCHOLARLY ACCOMPLISHMENTS

IV.i Holographic Measurements of Mean Inner Potential

This part of the research concentrated on measurement of mean inner potential of spherical particles (~30-100nm dia.). Polystyrene homopolymer and polystyrene/poly(acrylonitrile) latexes were studied as well as to amorphous and crystalline silicon. The following results were achieved.

- A value for Φ_o^{PS} of $8.2 \pm 0.3V$ was measured using 30-50nm diameter polystyrene latex made at Lehigh University by Drs. Olga Shaffer and Victoria Demonie. This can be compared to a pro-crystal value of 7-9V. A typical example hologram and its reconstructed phase and amplitude images is shown in fig. 2.

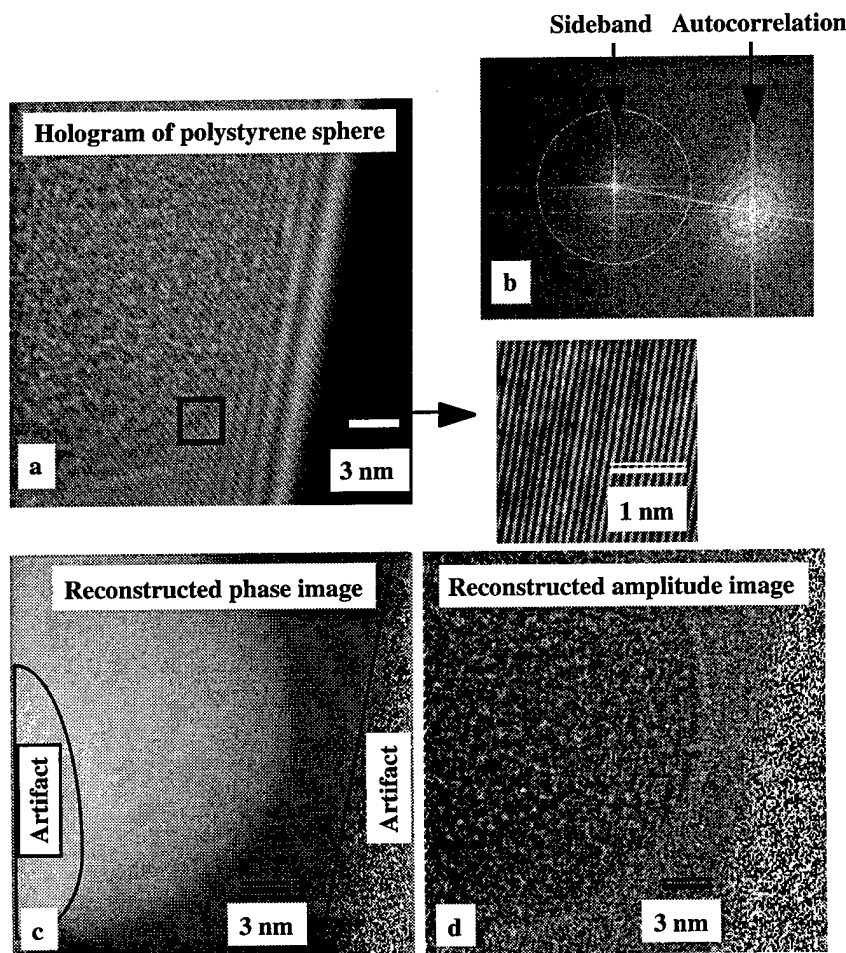


Fig. 2 - (a) electron hologram of a ~30 nm PS latex particle (b) FFT showing autocorrelation function and one sideband (c) reconstructed phase image (d) reconstructed amplitude image.

- First measurements of $\Phi_0^{\text{PS-PAN}}$ find a value of approximately 10V for a random polystyrene-polyacrylonitrile copolymer latex (40%PAN; provided by Lehigh). Additional work is ongoing to refine and interpret this measurement.

- Because polymer latexes are made via emulsion polymerization involving surfactants, simulations were performed to establish the effect of a thin surface layer of surfactant with different refractive properties than the bulk. The simulations were tested using 20nm-diameter amorphous Si nanospheres made by Prof. Thomas Kelly at the Univ. of Wisconsin Madison. These had a surface oxide layer of ~2nm thickness which could be distinguished from bulk Si using Stevens' analysis procedures. This experimental result is illustrated by figure 3.

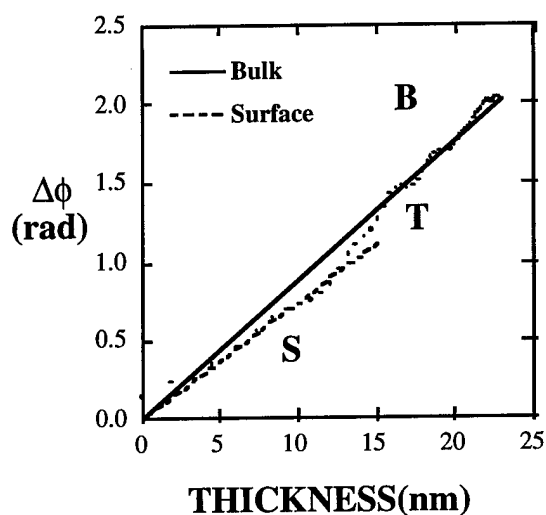
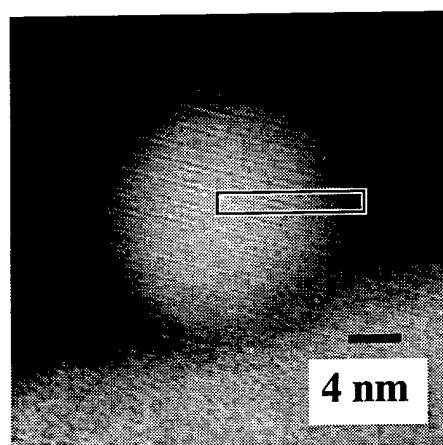


Figure 3 - (a) reconstructed phase image from a hologram of an amorphous Si nanosphere; (b) profile of phase shift as a function of position across the Si sphere in (a) where the oxide surface layer can be distinguished from the bulk Si by the change in slope ($d\phi/dt$).

- Φ_0 characteristic of amorphous silicon and crystalline silicon were determined as $11.9 \pm 0.3\text{V}$ and $12.1 \pm 0.4\text{V}$, respectively. The crystalline Si value agrees extremely well with recent first-principles LAPW calculations by Kim, Zuo, and Spence at ASU (in press, 1998). The Stevens measurement resolves some controversy over the value of Φ_0^{Si} raised by Gadgardiska et al. (Ultramicroscopy, 1994).

- Careful examination of both high-resolution (small field-of-view) and low-resolution (large field-of-view) holograms combined with simulations indicate that small (30-300nm diameter) polystyrene spheres do not develop measurable amounts of electrostatic charge during electron holographic observation. This is at odds with observations by several groups (Matteucci et al.; Frost et al.; Tanji et al.) who found charging in larger (1-5 μm diameter) latex spheres of polystyrene. Our observation is consistent with anecdotal evidence that small insulating particles do not charge under electron irradiation and suggests that there may be a critical size, determined by a characteristic electron diffusion length, below which charging does not occur.

IV.ii Phase-contrast and amplitude-contrast imaging of mesoscale structure in block copolymer thin films

A second portion of work concentrated on the development of microstructure in model block copolymers. This thread of research was initiated to create model 2-phase microstructures. Initially the work concentrated on the use of heavy-element stains to induce contrast. Subsequent and ongoing studies exploit these microstructures for holographic phase-contrast imaging experiments where stains are not used.

Thin-film specimens of SBS triblock copolymer were prepared by solution casting. The polymer was 30wt% polystyrene with $M_w=112,000$ and contained small amounts ($\leq 5\%$) PS/PB diblock and PS homopolymer. It was dissolved in toluene to produce a 0.1wt% solution. ~50 ml of solution was deposited on a water-polished NaCl single-crystal substrate in a small evaporation dish. The solvent evaporation rate could be controlled by adding toluene drops to the dish around, but not on, the substrate and by covering or partially covering the dish. Four different solvent evaporation rates were studied: fast (~200 nl/sec); intermediate (~5 nl/sec); slow (~1.5 nl/sec); and very slow (~0.2 nl/sec). Films were either studied in the as-evaporated condition or after annealing in vacuum (140°C, 18 hrs.). Films were stained by exposure to OsO_4 vapor and studied by TEM bright-field imaging.

These experiments led to the following conclusions:

- Under conditions where the films are exposed to solvent for long periods of time and are subjected to post-evaporation annealing, the morphology consists of PS cylinders lying in the

film plane arranged in a hexagonal array within a PB matrix. This morphology is expected from thermodynamic considerations.

- Several different film morphologies are observed as a function of solvent evaporation rate. These follow a logical progression from the least to the most ordered as the polymer mobility is increased via slower solvent evaporation (figure 5). Fast evaporation leads to a microphase-separated morphology where the domains have no long-range order. Slower evaporation rates lead to highly ordered morphologies of PS cylinders in a PB matrix. The cylinders can be oriented either in the plane of the film or perpendicular to it depending on the solvent evaporation rate.

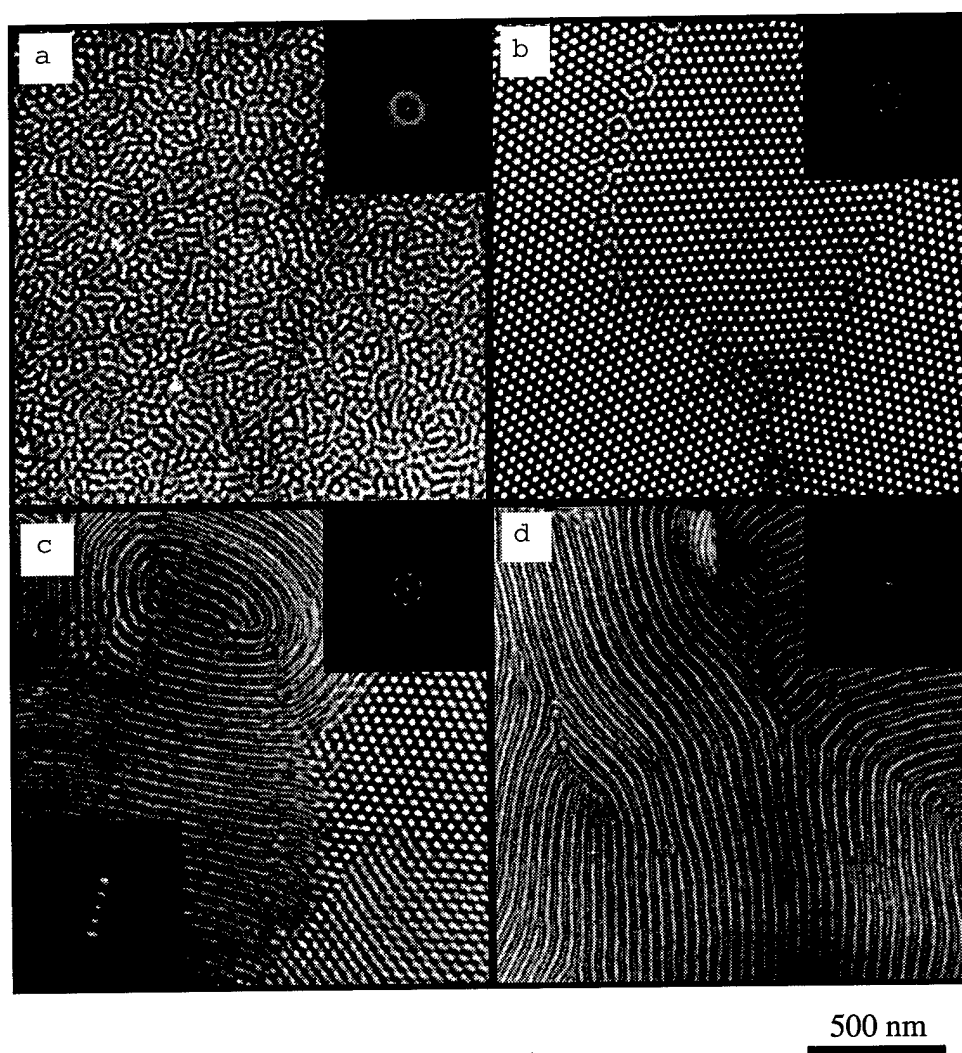


Figure 5 - Plan-view bright-field TEM images of the as-cast OsO_4 -stained SBS thin films with inset Fourier transforms:
(a) fast (~ 200 nl/sec); (b) intermediate (~ 5 nl/sec);
(c) slow (~ 1.5 nl/sec); and (d) very slow (~ 0.2 nl/sec).

- The array of in-plane cylinders in as-cast films shows a distorted hexagonal symmetry (fig. 6) which is attributed to an anisotropic contraction of the PB during solvent evaporation.

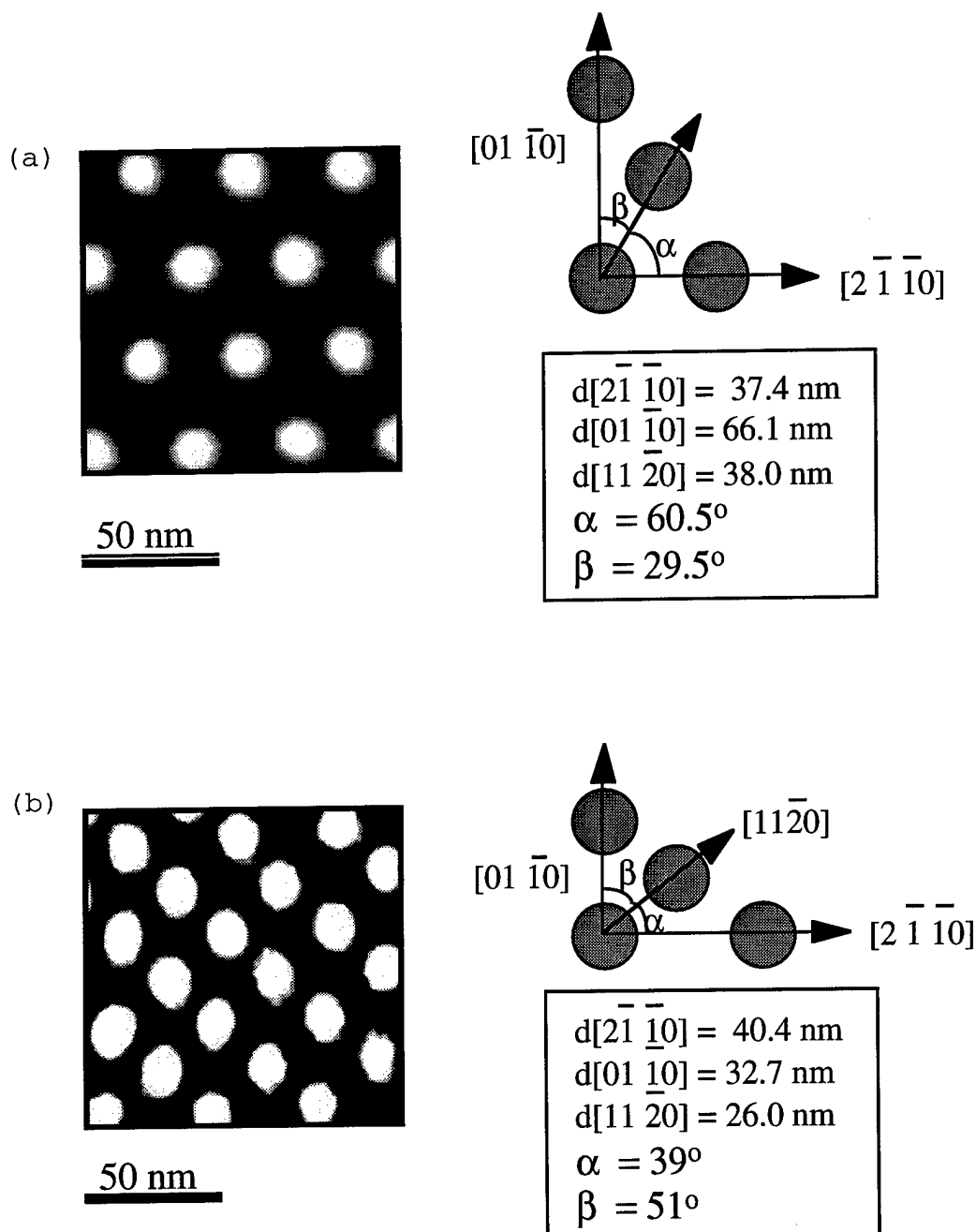


Figure 6 - (a) plan-view image of an as-cast film with the vertical-cylinder morphology and a schematic indicating a well-ordered hexagonal lattice; (b) cross-sectional image of ~300nm thick as-cast film with an in-plane cylinder morphology and a schematic indicating a distorted hexagonal lattice.

- Post-evaporation annealing either increases the interdomain spacing or transforms the as-cast microstructure into a different microstructure altogether. The coarsening effect is consistent with reports suggesting that kinetic constraints at low solvent concentrations prevent the microstructure from achieving its equilibrium interdomain spacing. Observations concerning morphological transformations suggest that nucleation of domains with a different morphology than the parent morphology is relatively difficult, but, once nucleated, growth of new domains proceeds with less of an activation barrier.

V. PUBLICATIONS

Archival Manuscripts

Measurement of polystyrene mean inner potential by transmission electron holography, Y.C. Wang, T.M. Chou, M. Libera, E. Voelkl, and B. Frost, *Microscopy and Microanalysis* (1998) in press.

Kinetic constraints on the development of surface microstructure in SBS triblock copolymer thin films, G. Kim and M. Libera, *Macromolecules* (1998) in press.

Microstructural development in solvent-cast polystyrene-polybutadiene-polystyrene (SBS) triblock copolymer thin films, G. Kim and M. Libera, *Macromolecules* (1998) in press.

Analysis and simulation of digital electron holograms of polymer latexes, Y.C. Wang, T.M. Chou, and M. Libera, *Scanning Microscopy Supplement 11* (1998), in press.

Energy-loss measurements of polymer microstructure and polymer interfaces: issues and opportunities, K. Siangchaew and M. Libera, *Microscopy and Microanalysis* 3, 530-539 (1997).

Transmission electron holography of silicon nanospheres with surface oxide layers, Y.C. Wang, T.M. Chou, M. Libera, and T.F. Kelly, *Applied Physics Letters* 10, 1296-1298 (1997).

Conference Proceedings

Measuring Polymer Microstructure Using Spatially-Resolved Energy-Loss Spectroscopy in the Scanning Transmission Electron Microscope, K. Siangchaew, D. Arayasantiparb, and M. Libera, in

Morphological Control in Multiphase Polymer Mixtures, ed. R. Briber et. al, MRS Symp. Proc. 461, (1997).

Energy-filtered imaging of polymer microstructure, K. Siangchaew, J. Bentley, and M. Libera, *Proceedings - Microscopy and Microanalysis 1996*, ed. G.W. Bailey et al. (San Francisco Press, 1996) 162-163.

Mean inner potential measurements of polymer latexes by transmission electron holography, Y.C. Wang and M. Libera, *Proceedings - Microscopy and Microanalysis 1996*, ed. G.W. Bailey (San Fran. Press, 1996) 168-169.

Cross-sectional TEM analysis of solvent-cast SBS thin films, G. Kim and M. Libera, *Proceedings - Microscopy and Microanalysis 1996*, ed. G. Bailey et al. (San Francisco Press, 1996) 1186-87.

On the Microstructure of the Epoxy/Adherend Interphase, M. Libera, W. Zukas, S. Wentworth, and A. Patel, *Polymer/Inorganic Interfaces*, ed. L.T. Drzal, R.L. Opila, N. Peppas, and C. Schutte, MRS Symp. Proc. V385, 65-70 (1995).

Transmission Electron Holography of Polymer Microstructure, M. Libera, J. Ott, and Y.C. Wang, *Electron Holography*, ed. by A. Tonomura, L. Allard, G. Pozzi, D. Joy, and Y. Ono (North Holland, Amsterdam, 1995) 231-238.

Morphological Transformations in Solution-Cast SBS Triblock Copolymer Thin Films" G. Kim, W. Marsillo, and M. Libera, *Proceedings Microscopy and Microanalysis*, Proceedings of the 53rd meeting of the Microscopy Society of America, ed. G.W. Bailey et. al, Jones and Begall, Boston, 1995, 184-185.

Effects of Carbon Fibers on the Crystallization of Nylon 6.6, K. Siangchaew, T. Davidson, and M. Libera, *Crystallization-Related Phenomena in Amorphous Materials*, ed. by M. Libera et al. (MRS Symposium Proc. V321, Pittsburgh, 1994) 651-656.

Off-Axis Holography of a Two-Phase Polymer, M. Libera, M. Gajdardziska, and M. Disko, in *Proc. 52 meeting Microscopy Society of America*, ed. G.W. Bailey and A.J. Garrett-Reed (San Francisco Press, 1994) 444-445.

VI. PRESENTATION OF PAPERS, SEMINARS, LECTURES, ETC.

The following list summarizes some of the principal presentations made during the course of this grant.

** indicates invited talk.

** Columbia University, New York, New York, *Seeing Polymer Microstructure*, 1/29/97.

** Lehigh University, Bethlehem, PA, *Electron refraction and phase contrast imaging of polymers*, 2/27/97.

** Hoechst-Celanese Research Center, Summit, NJ, *Using the electron microscope to measure polymer microstructure*, 1/16/97.

** Philadelphia Society for Electron Microscopy, Philadelphia, PA, *Seeing Polymer Microstructure*, 12/16/96.

** Rohm and Haas Corporation, Bristol, PA, *Seeing Polymer Microstructure*, 12/11/97.

Microscopy Society of America (Cleveland, OH), 8/97, *Electron refraction of amorphous nanospheres*

Materials Research Society (Boston, MA) 1997, *Measuring Polymer Microstructure Using Spatially-Resolved Energy-Loss Spectroscopy in the Scanning Transmission Electron Microscope; and The Distribution of SIS Compatibilizer in Melt-Mixed Polystyrene-Polyethylene Homopolymer Blends*.

Arizona State University Winter Workshop on Interfaces, Tempe, AZ, *Holography of nanospheres*, 1/8/97.

Arizona State University Workshop on Interfaces, Tempe, AZ, *Spectrum profiling of polymer interfaces*, 1/10/97.

** Hoechst-Celanese Research Center, Summit, New Jersey, *Seeing Polymer Microstructure*, 7/11/96.

** Allied-Signal Corporation, Morristown, NJ, 5/15/96, *Polymer microstructure studies using the electron microscope*.

** Lehigh University, Bethlehem, PA, Emulsion Polymers Institute, *Electron Holography of Polymer Latexes*," 5/2/96.

** University of Delaware, Newark, DE, 5/8/96, *Electron-optical techniques for studying polymer interfaces and interphases*.

** Microscopy Society of America, Minneapolis, MN, *Energy-filtered imaging of polymer microstructure*, 8/96

The following contributed papers were presented at the August 1996 meeting of the Microscopy Society of America (Minneapolis, MN) in collaboration with students and colleagues:

Mean inner potential measurements of polymer latexes by transmission electron holography.

Cross-sectional TEM analysis of solvent-cast SBS thin films.

Quantitative energy-filtered TEM imaging of interfaces.

Materials Research Society fall meeting, Boston, MA, contributed poster presentation, *Morphological transformations in self-assembled SBS triblock solution cast thin films*, 11/29/96.

15th Pfefferkorn Workshop on Digital Image Processing, Lake George, NY, *Transmission electron holography of polymer latexes*, contributed oral presentation, 5/22/96

7th KSEA Northeast regional conference, Rutgers Univ, Piscataway, NJ, *Transmission electron microscopy of compatibilizer distribution in PS and PE homopolymer blends*, 3/23/96.

** University of Wisconsin, Madison, *Transmission electron holography of polymer microstructure*, 12/14/95.

** Army Materials Technology Laboratory Watertown, MA, *Electron-optical imaging methods and their application to polymer microstructure*, 3/24/95.

** Stevens Physics Colloquium, Hoboken, NJ, *Transmission electron holography*, 10/5/97

** National Institutes for Standards and Technology, Gaithersburg, MD, *Electron holography in the transmission electron microscope*, 9/15/94.

Off-Axis Electron Holography of a Two-Phase Polymer, contributed talk, annual meeting of the Microscopy Society of America, New Orleans, 8/94.

Transmission Electron Holography of Polymer Microstructure, poster presented at the First International Workshop on Electron Holography, Knoxville, Tennessee 8/94.

VII. PERSONNEL SUPPORTED

Dr. Young-Chung Wang "Transmission Electron Holography of Polymer Microstructure," Ph.D. awarded May, 1997. Awarded departmental Traum prize for excellence in his Ph.D. studies. Currently working as a post-doc at the National Center for Electron Microscopy at Lawrence Berkeley National Laboratory.

Dr. Ginam Kim "The development of bulk and surface microstructure in solvent-cast SBS thin films," Ph.D. anticipated 31 September, 1997. Dr. Kim is now a post-doc with the polymer division at the National Institutes for Standards and Technology (NIST).

Mr. William Marsillo, Senior Design Project, B.S. awarded 6/94, "TEM Study of Polymer Morphology," recipient of an Undergraduate Research Scholarship from the Microscopy Society of America.

Mr. Krisda Siangchaew "Spectrum Profiling of Polymer Interfaces," Ph.D. anticipated August, 1998.

Mr. Alex Chou, "Quantitative phase contrast imaging of polymers by electron holography," Ph.D. anticipated May, 2000.

Prof. M. Libera: Principal Investigator, partial summer support.

VIII. INVENTIONS

None